Pre-Laboratory
Assignment

A Sequence of Chemical Reactions

A Cycle of Chemical Reactions of Copper

Complete these Pre-lab Questions.

Complete these exercises after reading the experiment but before coming to the laboratory to do it.

1. Write balanced chemical equations to represent each of the steps in the sequence of reactions performed in this experiment. Indicate the physical state of all reactants and products by writing (aq), (s), (l), or (g) after each formula.

2. Imagine that you start with exactly 1.00 g of copper. Determine the theoretical yield (in grams) of the copper-containing compound present at the end of each step in the above sequence.

3. In Part II of the experiment you dissolve $Cu(OH)_2$*(s)* in 3.0 M H_2SO_4*(aq)*. Calculate the quantity of H_2SO_4 required to react with 0.64 g of $Cu(OH)_2$ according to the equation you wrote in question one.

A Sequence of Chemical Reactions

A Cycle of Chemical Reactions of Copper

Learning Objectives

Chemistry concepts
Chemistry of copper and grouping the types of chemical reactions using a simple classification scheme
Lab techniques
Observing and recording chemical reactions and interpreting them in terms of chemical equations
Quantitative laboratory techniques including synthesis, filtration, separation and quantitative transfers

Background

The overall copper cycle is given in manual as

$$Cu(NO_3)_2\ (aq) \rightarrow Cu(OH)_2\ (s) \rightarrow CuSO_4\ (aq) \rightarrow Cu_3(PO_4)_2\ (s) \rightarrow CuCl_2\ (aq)$$

However, due to time restrictions we will shorten the cycle as

$$Cu(NO_3)_2\ (aq) \rightarrow Cu(OH)_2\ (s) \rightarrow CuSO_4\ (aq) \rightarrow Cu\ (s)$$

The balanced equations corresponding to these steps are:

1. Many transition metals form insoluble hydroxides which can be precipitated from solutions by soluble hydroxides such as NaOH. This is a **precipitation reaction**, which two soluble substances react to form an insoluble compound.

$$Cu(NO_3)_2\ (aq) + 2NaOH\ (aq) \rightarrow Cu(OH)_2\ (s) + 2NaNO_3\ (aq) \qquad \textbf{Eqn. (1)}$$

2. Metal hydroxides are bases which react with acid to form salt and water. This is an example of **acid-base reaction** where an acid + base goes to salt + water.

$$Cu(OH)_2\ (s) + H_2SO_4\ (aq) \rightarrow CuSO_4\ (aq) + 2H_2O\ (l) \qquad \textbf{Eqn. (2)}$$

3. Relatively more chemically reactive metals, such as magnesium, readily displace less reactive metals from their salts. This is often called a **displacement reaction.**

$$CuSO_4(aq) + Mg\ (s) \rightarrow MgSO_4(aq) + Cu\ (s) \qquad \textbf{Eqn. (3)}$$

This is a type of reaction called **oxidation-reduction reaction** which involves a transfer of electrons from the more reactive metal to the less reactive metal. This can be seen as changes in oxidation number of different chemical species involved. In **Eqn 3**, Mg(s) is oxidized to Mg^{2+} and Cu^{2+} is reduced to Cu(s).

To remove the excess Mg(*s*) left over from **Eqn. (3)** HCl(aq) is added which selectively oxidizes the more active Mg metal without affecting the less active Cu metal:

$$\mathrm{Mg\ (s)} + 2\mathrm{HCl}\ (aq)\ \rightarrow \mathrm{MgCl_2}\ (aq) + \mathrm{H_2}\ (g) \qquad \textbf{Eqn. (4)}$$

Purpose

You are to observe a series of reactions that convert copper from one chemical state to another. You are to practice quantitative laboratory techniques by recovery the copper with minima loss. In addition, you are to determine the actual percentage yield in the overall process by taking the actual mass of Cu(s) at end of experiment divided by the calculated mass of Cu from the initial $Cu(NO_3)_2$ solution multiplied by 100%.

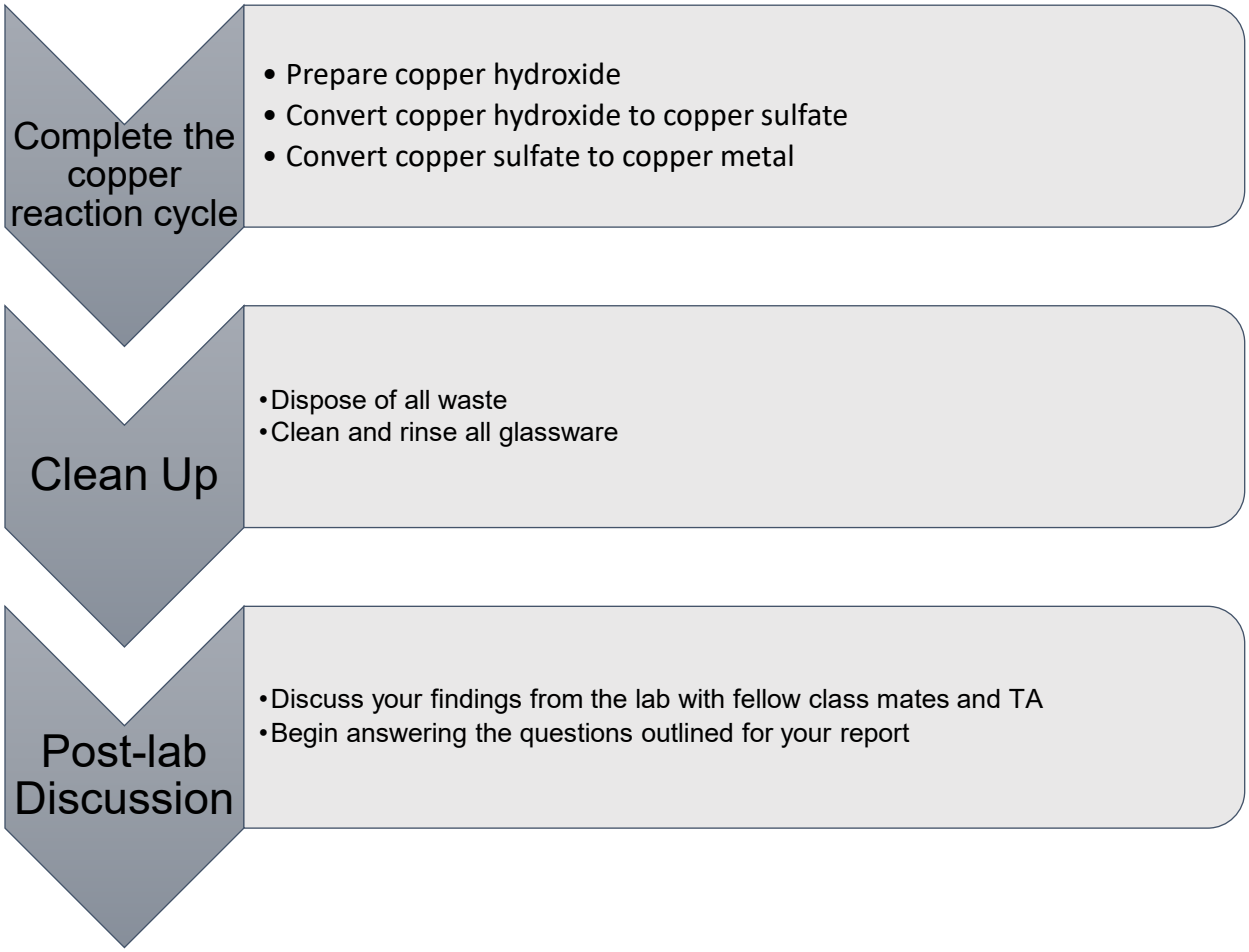
Procedure

⚠ Safety

Safety goggles must be worn at all times in the laboratory. The various acids used in preparing the copper solutions have different concentrations. Wear your gloves when handling the acids. The following safety issues must also be observed in the experiment.

- ✓ Sodium hydroxide solution (6 M) is corrosive and hazardous. It is especially hazardous to eyes. Wear gloves while handling and avoid fumes when handling. Wash your hands carefully after handling it, even if you think you have not gotten any on your skin.
- ✓ Sulfuric acid (H_2SO_4 3.0 M) is corrosive and hazardous. Wear gloves and avoid fumes by keeping face away while handling this acid.
- ✓ Concentrated HCl (6 M and 12 M) is corrosive and hazardous. Wear gloves and avoid fumes by keeping face away while handling concentrated HCl.
- ✓ Hydrogen gas is very flammable. Work only in the hood.
- ✓ Acetone is flammable. Keep away from hot plates and flames.

Procedural Diagram



Part I. Preparation of Copper(II) Hydroxide

Obtain a solution of copper (II) nitrate from the stock solution in the room. ***Record the concentration of the solution and its color in your notebook*.** Measure out 25.00 mL of the solution. Place the solution in an ice or water bath.

To the homogenous solution of copper(II) nitrate add 2 mL of concentrated NaOH (6 M, where the "M" refers to the concentration of the substance, in units of moles per liter), drop-wise with constant stirring. The blue gelatinous precipitate that forms is copper(II) hydroxide, $Cu(OH)_{2(s)}$. Let the mixture settle and use litmus paper (as directed below) to test the acidity of the supernatant liquid (the liquid sitting above the settled precipitate). *This is necessary because the precipitation of $Cu(OH)_{2(s)}$ is not complete until the supernatant liquid is basic.*

To use litmus paper to test whether a solution is acidic or basic, place a piece of red and a piece of blue litmus paper on a clean watch glass, dip a glass stirring rod into the solution, withdraw it,

and touch the moist tip to each kind of litmus paper. **Acid turns blue litmus red, and base turns red litmus blue.** In this case the gelatinous blue precipitate can, if it is stirred up, mimic the blue of the litmus indicator. So let precipitate settle before a litmus paper test is to be done. If some precipitate does transfer to litmus paper, check the color of the litmus paper only at the very edge of the moist paper area where the solution spreads out on the paper without precipitate.

Add additional 10-drop portions of 6 M NaOH with stirring as necessary until the solution is just barely basic to litmus paper.

Once the solution is basic, add 30 mL of water to the beaker, stir well, and allow the mixture to stand once again for a few minutes while the precipitate settles. Carefully **decant** (pour off) the major part of the clear, colorless supernatant liquid into a beaker. Be certain not to pour off any of the precipitate.

Wash the precipitate with about 50 mL of water. To do this, add 50 mL of de-ionized water to the blue solid, stir well, and allow the precipitate to settle. Pour off (decant) most of the clear, supernatant liquid, always taking care not to lose any of the precipitate. Repeat with a second 50 mL portion of de-ionized water. Save the precipitate and the residual liquid in which it is suspended for Part II. Flush the washings down the drain with plenty of water. ***Record your observations of the reaction when 6 M NaOH to the is added to your $Cu(NO_3)_2$ solution in your notebook.***

Part II. Preparation of Aqueous Copper(II) Sulfate

With the $Cu(OH)_2$ precipitate sitting on your ice, slowly add dilute (3.0 M) H_2SO_4, drop-wise, with constant stirring, to the beaker containing the suspension of $Cu(OH)_2$ prepared in Part I. This needs to be done slowly so reaction is not over-heated and excess acid is avoided. Continue until the last bit of precipitate is dissolved. Give the precipitate time to dissolve rather than trying to hurry things by adding large amounts of acid. ***Record in your notebook the details of any color changes that you see.*** Save the colored solution of $CuSO_4(aq)$ for Part III.

Part III. Preparation of Copper Metal

In this step the aqueous Cu^{2+} ions from $CuSO_4(aq)$ will be reduced to copper metal by magnesium metal. This is an oxidation-reduction reaction in which the more reactive metal, magnesium, displaces the less reactive metal, copper, from copper(II) sulfate solution. Any excess magnesium is dissolved by the hydrochloric acid that is present, liberating hydrogen. Because copper is less reactive than hydrogen, it is not acted upon by the hydrochloric acid.

To the solution of $CuSO_4(aq)$ outside the ice-bath, add 4 or 5 pieces of magnesium turning and keep them submerged with the tip of a glass stirring rod. Have an ice-bath ready to moderate the vigor of the reaction, if necessary. As the $Cu^{2+}(aq)$ ions in the $CuSO_4$ solution is "reduced" by magnesium metal, Cu will precipitate out and the color of the solution will gradually disappear as Cu^{2+} ions are consumed. During the reaction some magnesium reacts with residual H2SO4 from previous step which also liberates hydrogen gas as a side-reaction. This by-product gas is flammable so allow no flames in the vicinity of this reaction.

If necessary, add more pieces of magnesium to until the color become clear. Finally, add the minimum possible quantity of concentrated (12 M) HCl, drop-wise, to consume any excess magnesium remaining. If there is no excess magnesium, then no HCl is needed.

When the color of the solution has disappeared and no copper forms at the surface of a small test-piece of magnesium dipped into the solution, and when the magnesium is completely dissolved, the reaction is over. There should be a quantity of brown-red particles of copper suspended in a colorless solution. Allow the copper to settle. ***Record your observations of the reaction with magnesium. Record your observations of the final copper metal product.*** Decant and discard the major part of the supernatant liquid and wash the solid copper dust four times with 15 mL portions of de-ionized water.

Decant as much water as possible from the beaker leaving behind all the copper. To dry the copper, wash it with small portions of acetone. This is done by adding a just enough acetone to cover the copper metal and then decant the acetone as done with water before leaving all copper behind. The acetone washes away the water which adheres to the copper dust. Acetone is much more volatile than water which means it evaporates much faster due to its higher vapor pressure. After acetone has replaced water in adhering to the copper dust, it takes only a matter of minutes for any residual acetone to evaporate away and leave dry copper metal product. Do not heat the sample and keep all sources of heat away from acetone because acetone is flammable.

The Final Step

Take a small square of weighing paper (or small clean, dry beaker) and weigh it on analytical balance. ***Record the mass in your notebook.*** Then with weighing paper on a watch glass (or pre-weighed beaker) and carefully transfer all of the dried copper onto the pre-weighed weighing paper (or into pre-weighed beaker) by judicious combination of tapping and scraping. Take the watch glass with weighing paper and copper (or pre-weighed beaker with copper) back to the same analytical balance and carefully transfer the weighing paper with copper metal without watch glass (or pre-weighed beaker with copper) to the mass balance pan. ***Measure and record mass of weighing paper with your dried copper to determine the mass of copper at the end of your reaction sequence.*** The mass of copper obtained from the cycle of reactions equals mass of the weighing paper (or pre-weighed beaker) with copper minus mass of weighing paper alone (or beaker) measured previously. Compute your percent yield of copper.

Clean Up

- ✓ **All aqueous solutions may be poured down the drain.**
- ✓ **All glassware should be rinsed and cleaned with water.**
- ✓ **Return all glassware to their designated location at the end of the lab period.**

Answer following Postlab Questions

1. Suppose that you start with 0.303 g of copper wire and faithfully follow the procedure. At the end you find that the copper dust weighs 0.309 g. Based on your understanding of the experimental procedure, offer at least two rational explanations for this apparent violation of the law of conservation of mass.
2. At the end of the reaction sequence, Mg is added to the copper-containing solution to form copper metal. How do you know when no more dissolved copper is in the solution? How can you test to see there is no more excess magnesium?
3. Based on what you saw as you performed the experiment, what practical obstacles are there to weighing the copper metal to check its actual yield?